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Glass Fiber and Nanoclay Reinforced Polypropylene Composites: Morphological, Thermal and Mechanical Properties

(Polipropilena Diperkuat dengan Gentian Kaca dan Nanotanahliat: Sifat Morfologi, Terma dan Mekanik)

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ABSTRACT

Hybrid composites of polypropylene (PP)/nanoclay (NC)/glass fiber (GF) were prepared by extrusion and injection molding. Molded specimens were analyzed by transmission electron microscopy (TEM), thermogravimetric analysis (TGA), tensile and flexural tests. TEM results revealed NC particle intercalation. TGA results showed that the incorporation of clay into the GF composite improves the thermal stability of the material. The initial thermal decomposition temperatures also shifted to higher values. Incorporation of GF into PP lowers the tensile strength of the binary composite, indicating poor fiber-matrix interfacial adhesion. However, introducing NC increased the strength of the ternary composites. Tensile modulus was enhanced with the incorporation of GF and further increased with an introduction of NC. Flexural strength and flexural modulus are both enhanced with an increase in GF and NC loading.

Keywords: Hybrid composites; mechanical property; nanostructured materials; thermal property

ABSTRAK

Bahan komposit hibrid polipropilena (PP) / tanah liat (TL) / gentian kaca (GK) disediakan dengan menggunakan ekstrusi dan acuan suntikan. Sifat morfologi bahan komposit acuan suntikan dikaji menggunakan teknik TEM. Kestabilan terma bahan komposit dianalisis menggunakan teknik TGA, manakala ciri-ciri mekanikal komposit dikaji dengan menggunakan ujian regangan dan lenturan. Analisis TGA menunjukkan bahawa penambahan TL ke dalam komposit yang mengandungi GK meningkatkan kestabilan terma bahan tersebut. Selain itu, suhu penguraian peringkat awal bahan komposit juga didapati beranjak ke nilai yang lebih tinggi. Penambahan GK ke dalam PP didapati menyebabkan penurunan nilai kekuatan regangan komposit perduaan, menunjukkan bahawa interaksi antara muka diantara GK dan PP adalah lemah. Walau bagaimanapun, kekuatan regangan komposit pertigaan menunjukkan peningkatan dengan penambahan TL ke dalam sistem. Penambahan GK meningkatkan modulus regangan komposit dan nilai ini bertambah dengan kehadiran TL ke dalam sistem. Kekuatan dan modulus bagi ujian lenturan didapati meningkat dengan penambahan GK dan TL ke dalam bahan komposit.

Kata kunci: Bahan berstruktur nano; komposit hibrid; sifat mekanik; sifat terma

INTRODUCTION

Polypropylene (PP) is one of the excellent thermoplastic polymers, characterized by its outstanding cost-to-performance ratio. The commercial significance of PP has resulted in an ever increasing drive to enhance its properties further, in particular through reinforcement with various particulates, fibers and layered inorganic fillers (Chen et al. 1998; Hong et al. 2008; Vilaseca et al. 2010). The effectiveness of inorganic fillers in improving the physical and mechanical properties of PP strongly depends on the filler aspect ratio, size, shape, surface characteristics, interfacial adhesion and degree of filler dispersion (Jordan et al. 2005). It has repeatedly been shown that an inorganic filler such as glass fiber increases the tensile modulus of such composites, yet causes the decrease of the strength and toughness. This could be due to the stress concentration, poor fiber-matrix adhesion and confinement of the matrix molecular mobility around the rigid filler phase.

A relatively new development in the field of polymer composites is the introduction of clay nanoparticles (1 – 100 nm), in which small quantities of high-modulus nanoclay (NC) have been shown to give significant improvements in mechanical, barrier and thermal properties (Modesti et al. 2006; Samal et al. 2008). Although numerous researchers have investigated individually PP/GF composites and PP/NC composite systems, only a few investigations have been reported on PP/GF/NC composites.

Mohan and Kanny (2011) in their work, combined PP nanocomposites matrix with chopped GF using a single screw extruder in a one–stage compounding. A small amount (5% fiber weight fraction, W_f) of nanoscale dispersed layered silicate was shown to enhance the degree of crystallinity and tensile properties as well as rheological and wear properties of the composites. In addition, Chandradass et al. (2008) used 3% W_f nanofillers in a GF reinforced vinyl ester matrix and studied the mechanical,

dynamic mechanical and vibrational properties. In both cases, the investigated properties were found to improve relative to the neat matrix and glass fiber composites.

However, in the present work, PP/clay nanocomposite systems were prepared for use as a matrix material for GF composites using a twin screw extruder in a two–stage compounding. An experimental study was carried out to exploit the functional advantages and potential synergistic effect on GF and NC in order to enhance the overall properties of PP. As a comparison, the properties of PP/GF composites and PP/NC nanocomposites were also evaluated under identical test conditions. Morphological, thermal and mechanical properties of the composites were investigated by transmission electron microscopy (TEM), thermogravimetric analysis (TGA), tensile and flexural tests, respectively.

Materials and Methods

PP (Propelinas H022) was supplied by Petronas, Malaysia. The neat PP was in the form of pellets with a melt flow index of 11 g/10 min, melting temperature of 165°C and a density of 910 kg.m^{-3}. Chopped E-glass fiber, surface-treated with silane and having a density of 2550 kg.m^{-3}, average diameter of 14 μm and length of 6 mm was obtained from KCC Corporation, Korea and used as the principal reinforcement. The nanoclay (type PGV) is a natural untreated montmorillonite clay, with a density of 776 kg.m^{-3} and a particle size of about 16 μm and manufactured by Nanocor, USA.

PP/GF and PP/NC composites were prepared with different ratios of PP/GF and PP/clay powder, respectively, as presented in Table 1. Compositions were physically pre-mixed and then compounded using a Brabender, KETSE 20/40 (Germany) twin screw extruder with the screw diameter and screw aspect ratio of 20 mm and 40, respectively. The temperature profile was set between 185°C and 190°C. For the PP/GF composites, the screw speed was set to 100 rpm, whereas for the PP/NC composites, the screw speed used was 800 rpm. The materials extruded from both formulations were pelletized into length of about 6 mm. In order to produce PP/GF/NC composites, the different ratios of the PP/NC and GF were physically mixed and re-compounded in a twin screw extruder, using the same temperature profile and screw speed of 100 rpm. Dumb bell–shaped tensile test specimens, according to ASTM Standard D-638 (2003) were then injection molded using a Boy® 55M (Germany), with a 55–tonne clamping force injection molding machine. The processing temperature was set between 175°C and 185°C and the mould temperature was set at 25°C. The list and abbreviation of specimens prepared are given in Table 1. Specimens were designated according to their compositions: for example (PP85/G15)/NC3 was referred as specimen with 85 wt% of PP, 15 wt% of GF and 3 phr of NC.

The microstructure of the PP/NC and PP/GF/NC were analyzed with a Hitachi H-600 (Japan) transmission electron microscope (TEM). The samples were ultra-microtomed with a diamond knife on a Leica Ultracut UCT (UK) microtome at room temperature to give section with a nominal thickness of 200 nm. Sections were transferred to 400 mesh Cu grids. Bright-field TEM images of the composites were obtained at an acceleration voltage of 300 KeV.

Thermogravimetric (TGA) analysis was done using a TGA 6 thermogravimetric analyzer (Perkin Elmer) under nitrogen atmosphere with a flow rate of 20 mL.min^{-1} and a scan rate of 10°C.min^{-1} over a temperature range of 50°C to 850°C. A test sample of 5-10 mg was used for each run.

Tensile tests were carried out using a universal testing machine, Instron 5569 (50 kN load-cell) at a constant cross-head speed of 5 mm.min^{-1} at 25°C. The tensile properties were determined according to ASTM D-638 (2003). The composite modulus was recorded at 0.5% strain. Flexural tests were carried out using the same machine and following ASTM D-790 (2010). The distance between the support spans was 50 cm. All the specimens were tested at a constant cross-head speed of 1.28 mm.min^{-1}.

TABLE 1. Designation and compositions of composite specimens

Sample	Matrix weight fraction, W_m (%)	Fiber weight fraction, W_f (%)	Fiber volume fraction, V_f (%)	Clay content (phr)
PP	100	-	-	-
PP100/NC3	100	-	-	3
PP100/NC6	100	-	-	6
PP100/NC9	100	-	-	9
PP85/G15	85	15	6	-
PP70/G30	70	30	14	-
(PP85/G15)/NC3	85	15	6	3
(PP85/G15)/NC6	85	15	6	6
(PP85/G15)/NC9	85	15	6	9
(PP70/G30)/NC6	70	30	14	6

Results and Discussion

Transmission Electron Microscopy (TEM)

To gain an insight into the morphology of the nanocomposites, characterization using TEM was conducted for 3, 6 and 9 phr NC–based nanocomposite samples, as shown in Figure 1. Nanocomposites containing 3 – 9 phr of NC show heterogeneous dispersion, presence of aggregates, as well as resin-rich areas in the composites. However, there are regions where completely delaminated sheets are dispersed individually as shown from the average dark lines of 1 – 2 nm thickness (Figure 1(a)). The size of the aggregates and the number of platelets in the composites increase (Figure 1(a) – 1(c)) with the percentage of clay within the PP matrix. Hussain et al. (2007) suggested that the degree of intercalation or exfoliation depends on the type and surface modification of NC, presence of compatibilizing agent, as well as the processing condition. Within the scope of this study, untreated NC has been used. Therefore, a better dispersion of layered silicates is difficult to attain as the NC has a strong tendency to agglomerate and interaction between PP and NC may be poor as no compatibilizer has been added.

Thermogravimetric Analysis (TGA)

The thermal stability of the nanocomposites was studied by the TGA technique. Figures 2 and 3 show the TGA scans in the form of weight change and derivative weight change versus temperature for PP, PP/NC composites and PP/GF/NC hybrid composites. Table 2 presents the quantitative values of the onset, derivative peak temperature and the temperatures at 5%, 10% and 50% of weight loss, which are referred to as: T_{onset}, DT_p, $T_{5\%}$, $T_{10\%}$ and $T_{50\%}$, respectively. The TGA curves in Figure 2 demonstrate that the incorporation of clay in PP matrix gives insignificant improvement to the thermal stability of the material. T_{onset} of PP/NC composites (Table 2) are quite similar with the T_{onset} value recorded for PP matrix (355°C). On the other hand,

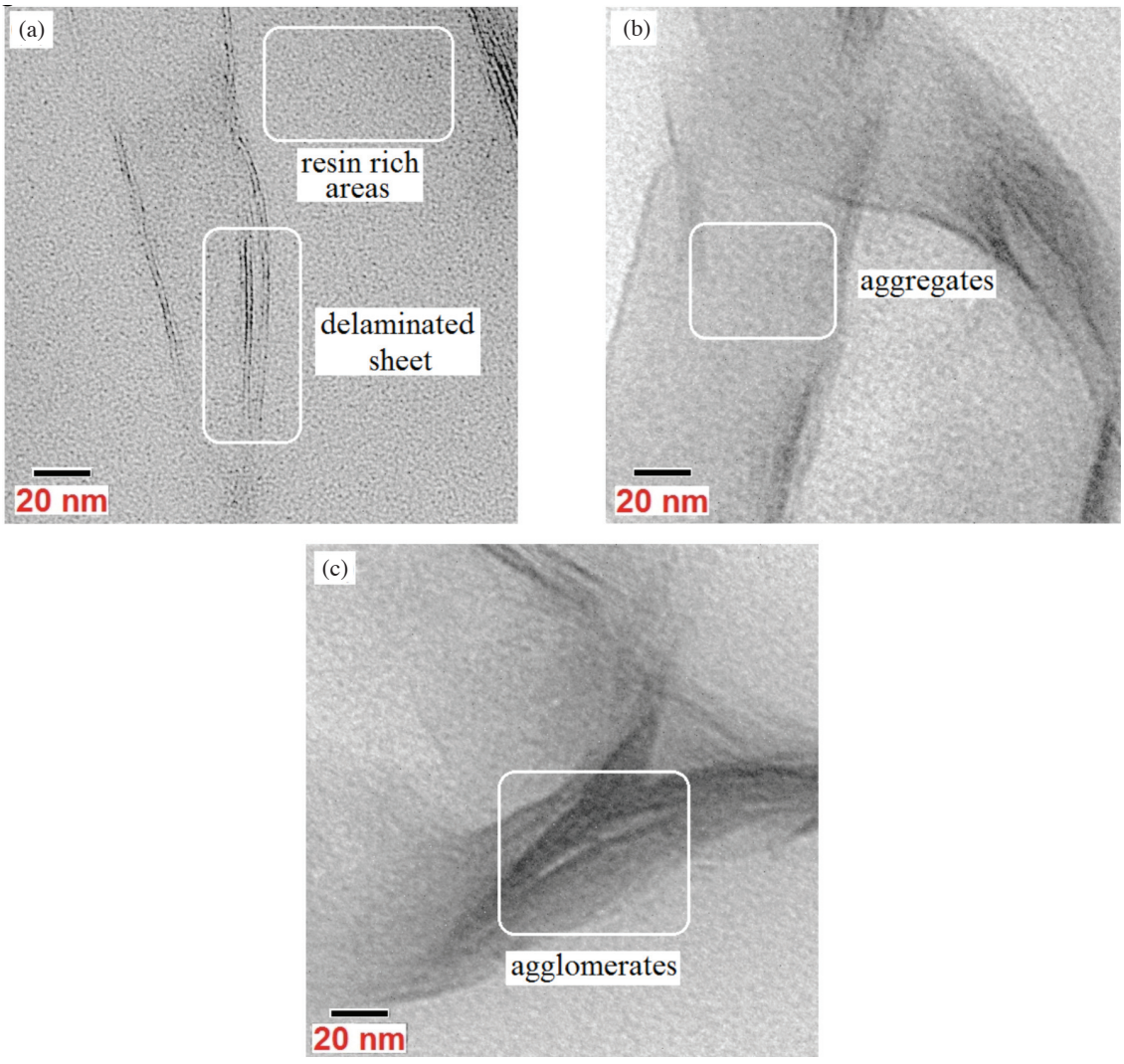

FIGURE 1. TEM images of PP/NC as a function of NC concentration: (a) PP/NC3; (b) PP/NC6 and (c) PP/NC9; at 97,000x magnification

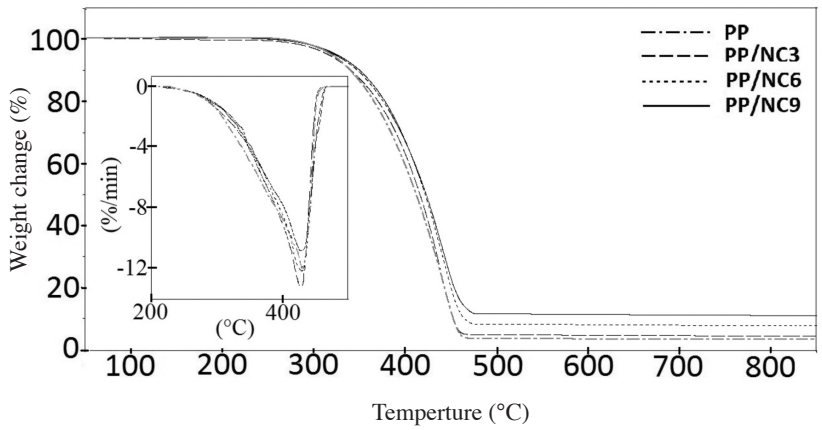

FIGURE 2. TGA thermograms of PP and PP/NC nanocomposites

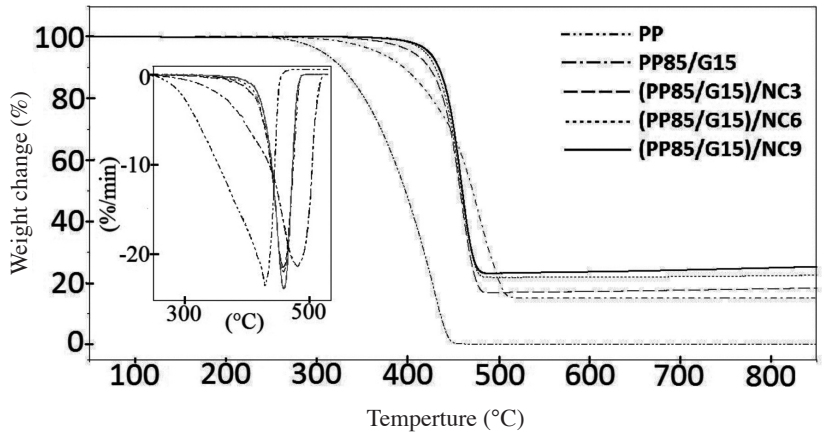

FIGURE 3. TGA thermograms of PP, PP/GF and PP/GF/NC hybrid composites

TABLE 2. TGA thermogravimetric data of PP and composites

Sample	Decomposition temperature range (°C)	T_{onset} (°C)	T_{onset} (Std. dev)	$T_{5\%}$ (°C)	$T_{10\%}$ (°C)	$T_{50\%}$ (°C)	DT_P (°C)
PP	231 - 466	355	2.6	307	328	397	431
PP100/NC3	223 - 459	367	2.5	303	328	401	427
PP100/NC6	209 - 466	363	2.4	312	337	407	430
PP100/NC9	210 - 469	363	3.0	310	334	408	429
PP85/G15	250 - 525	422	2.6	368	392	471	481
(PP85/G15)/NC3	321 - 491	431	2.0	405	424	458	458
(PP85/G15)/NC6	332 - 492	433	3.1	420	430	459	459
(PP85/G15)/NC9	354 - 494	436	2.0	424	435	461	460

the TGA curves for hybrid composites (Figure 3) shows that the incorporation of clay in GF composites improves the thermal stability of the material. Degradation takes place at higher temperature in the presence of clay. Incorporation of 15% W_f of GF increases T_{onset} to 422°C. Addition of 3 phr of clay into the composites further increases the T_{onset} of (PP/G15)NC to 431°C. There is no significant different in the T_{onset} on further addition of 6 and 9 phr of clay. This behavior can be attributed to the adsorption effect by the addition of clay, which limits the emission of the gaseous degradation products. Krump et al. (2006) and Duquesne et al. (2003) suggested that the volatile degradation products are adsorbed on the surface of the fillers, resulting in an increase in the thermal stability of the material.

It is expected (from Figure 1) that at higher clay content (6 and 9 phr), agglomerations of fillers should result in lower thermal stability (Lafia-Araga et al. 2012). However, the clay is thermally stable

within the experimental temperature used in this study. In other words, the thermal degradation of the matrix is the predominant process being observed. Therefore, irrespective of the amount of clay present and agglomeration being experienced, the thermal stability of the composites remains relatively the same.

The initial thermal stability is characterized by the temperatures at 5% and 10% weight changes ($T_{5\%}$ and $T_{10\%}$). The $T_{5\%}$ and $T_{10\%}$ values remain essentially unchanged as clay nanoparticles were added to the PP matrix (Figure 2 and Table 2). By contrast, it can be seen that these initial thermal decomposition temperatures are enhanced by the addition of clay into the GF composite. At 5% weight change, the $T_{5\%}$ increases from 368°C for PP/G15 to 405°C, 420°C and 424°C for composites containing 3, 6 and 9 phr NC, respectively. The same trend is observed at 10% weight change. At this initial degradation event, it is possible that the polarity of PP/GF composites is enhanced by the incorporation of NC. This synergy could lead to better compatibility, resulting in the observed trend (Figure 3). In addition, it is believed that a good dispersion of clay platelet can act as a barrier, trapping the volatilizing matrix from escaping into the atmosphere.

By acting as a heat barrier, these high aspect ratio fillers, which not only enhance the overall thermal stability of the system, but also promote char formation, resulting in a high performance carbonaceous silicate layer, which will insulate the underlying matrix material (Sharma & Nayak 2009). Furthermore, for hybrid composites (Figure 3), at temperatures higher than cross-over between degradation curves (447°C), the thermal behavior of GF becomes predominant and an increase in the thermal stability of the GF composite (PP/G15) is observed. This may explain why the values of DT_p and $T_{50\%}$ for PP/G15 are higher than those values for composites with increasing clay loading (Table 2).

TENSILE PROPERTIES

Tensile properties (tensile strength, modulus and strain) of PP/GF/NC composites with different levels of fiber and clay contents are shown in Figure 4 and 5. For PP/GF and PP/NC composites, the tensile strengths decrease as filler contents increase. For example, the tensile strength is reduced from 31 MPa for PP to 28 MPa for both PP/G30 and PP/NC9 composites. This reduction in strength is an indication of poor filler-matrix adhesion and lack of stress transfer capability of the fillers (Oksman et al. 2009; Thomason 2002). It is assumed that when there is poor adhesion at the interface at high deformations, as it happens in these tensile tests, the presence of fillers or fibers in a polymer matrix gives rise to defects at the interface, which is responsible for the strength reduction (Mohd Ishak et al. 2001).

However, the addition of 6 phr clay in 15% W_f, (PP/G15)/NC6 and 30% W_f, (PP/G30)/NC6 results in an increase in tensile strength by 10% and 6%, respectively, relative to PP/GF composite at the same fiber loading (Figure 4). It is possible that the presence of clay increases the interfacial adhesion between GF and PP, hence improving the tensile strength of the PP/GF/NC composite. In addition, the silane treatment of the GF could also have intensified the synergy between PP, GF and NC as enhanced coupling is expected.

From Figure 4, it can be seen that tensile modulus of composites are increased from 70% to 105%, for PP/G15 and PP/G30 composites, respectively, relative to the PP matrix. On the other hand, in the case of (PP/G15)/NC6 and (PP/G30)/NC6, the modulus increment is more distinct, by 100% to 112% upon GF addition, which is in accordance with the trend reported previously (Chandradass et al. 2008). It is revealed from Figure 4 that tensile modulus of PP/NC6 composite is increased by 50% (3.02 GPa) when compared with PP matrix (2.02 GPa). It has been reported by other researchers (Hussain et al. 1996) that

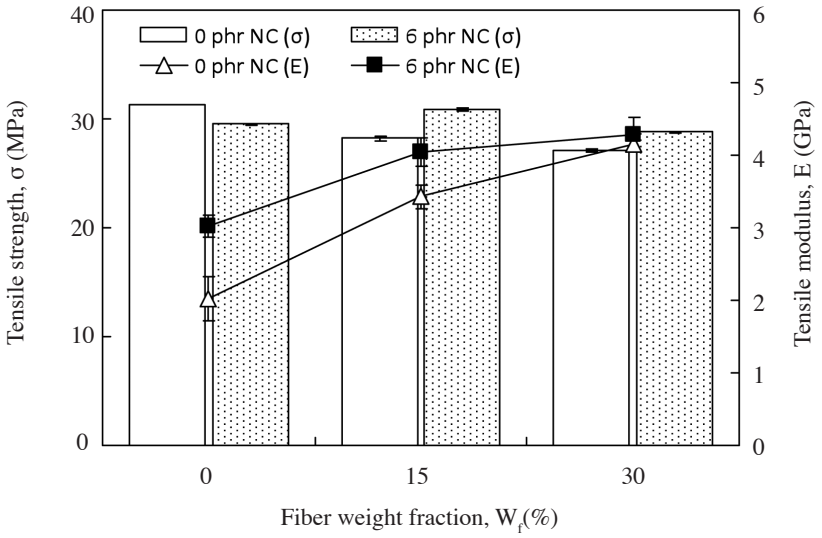

FIGURE 4. Tensile strength and tensile modulus of PP and composites

the improvement of modulus in the hybrid composites (PP/GF/NC) is mainly attributed to the improvement of the matrix modulus from particulate filler dispersion. Thus, it seems that a synergistic effect takes place by incorporating particulate filler into the matrix, leading to higher stiffness than would otherwise be expected, solely on the basis of the change in the matrix modulus. At the same fiber content, the incorporation of NC into the system further enhances the tensile modulus. Modulus of (PP/G15)/NC6 composite increases by about 18%, as clay is added to the system relative to PP/G15. However, at 30% W_f, the effect of NC addition on the tensile modulus is no longer significant. The same behavior has also been reported by Zhao et al. (2001). It has been pointed out that when the fibers exceed a certain loading level, the reinforcing efficiency of the fibers will begin to decrease (Hiscock & Bigg 1989; Lee & Jang 1999). At higher fiber loading, the probability of fiber to fiber contact is high. This is due to the inability of the matrix to completely encapsulate the fiber (wetting problem), resulting in negative reinforcement effect.

On the other hand, tensile strain (Figure 5) is simultaneously reduced with the incorporation of GF, which is attributed to the fact that reinforcing fibers strongly restrain the deformation of the matrix polymer, as demonstrated in several previous studies (Fu & Lauke 1998; Hassan et al. 2011; Thomason et al. 1996). The trend remains essentially unchanged as clay nanoparticles are added to the system, except that tensile strain decreases further.

Figure 6 shows tensile properties of 15% W_f GF composite as a function of NC content. It is observed that the tensile strength and tensile modulus of the composites generally increase with increasing clay content. The tensile strength increases slightly with filler content of 6 phr, beyond which it decreases slightly. An insignificant change (1% reduction) in tensile strength is observed with the addition of 3 phr of clay into PP/GF15 composite. Further addition of 6 phr clay in the matrix, (PP/G15)/NC6 increases the tensile strength by 10%. However, composites, with 9 phr of clay, (PP/G15)/NC9 show a slight decline in tensile strength. This could be due to agglomeration of NC in the composites with higher NC loading as can be seen in Figure 1, forming stress concentration areas, especially in tension, thus resulting in premature failure. Other researchers (Hussain et al. 1996; Wetzel et al. 2003) have also reported that the presence of agglomeration of filler in epoxy GF composites, obviously caused deterioration in the mechanical properties of the material.

It is observed that tensile modulus of hybrid composites increased with increasing clay content (Figure 6). An improvement in tensile modulus of about 22% is observed with an addition of 6 phr clay, relative to PP/G15 composite. The improvement of modulus could be due to the exfoliation of clay nanoparticles in the matrix that restricts the mobility of polymer chains under loading. However, by incorporating 9 phr clay into the composite, only 18% increment in tensile modulus is observed. It has been suggested that at higher clay contents, large agglomerates in the matrix may have occurred, leading to the reduction in tensile modulus (Wetzel et al. 2003). On the other hand, the tensile strain is simultaneously reduced with increase in clay particle content. The tensile strains are reduced from 7% for PP/G15 to 6% for (PP/G15)/NC3 and 4% each for (PP/G15)/NC6 and (PP/G15)/NC9. This can be attributed to the fact that clay particles strongly restrain the deformation of the composites as demonstrated by Asi (2009).

It has been mentioned earlier that a good dispersion results in better thermal stability. It also enhances better tensile properties. However, in the TGA results, the presence of clay in the hybrid system improved the thermal stability of the neat matrix irrespective of the clay content and the agglomeration being encountered (Table 2 and Figure 3).

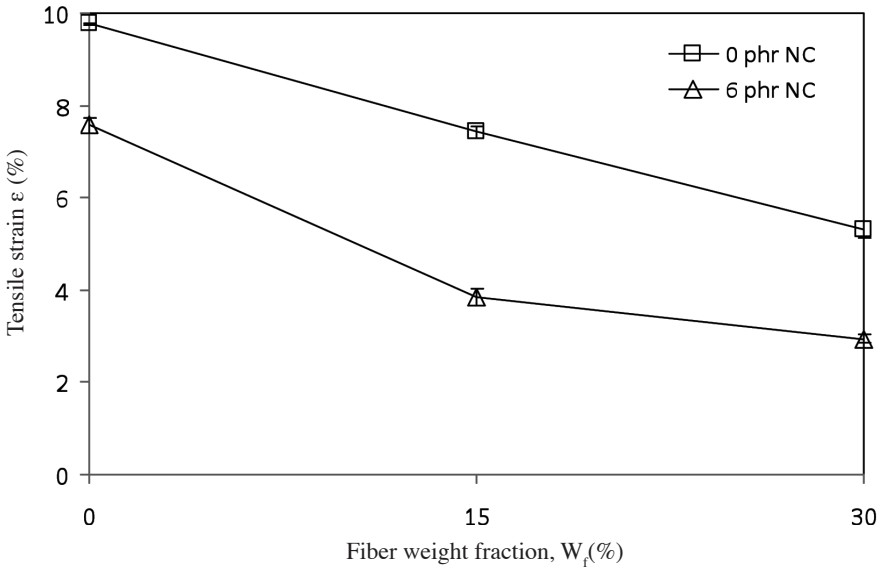

FIGURE 5. Tensile strain of PP/GF and PP/GF/NC hybrid composites

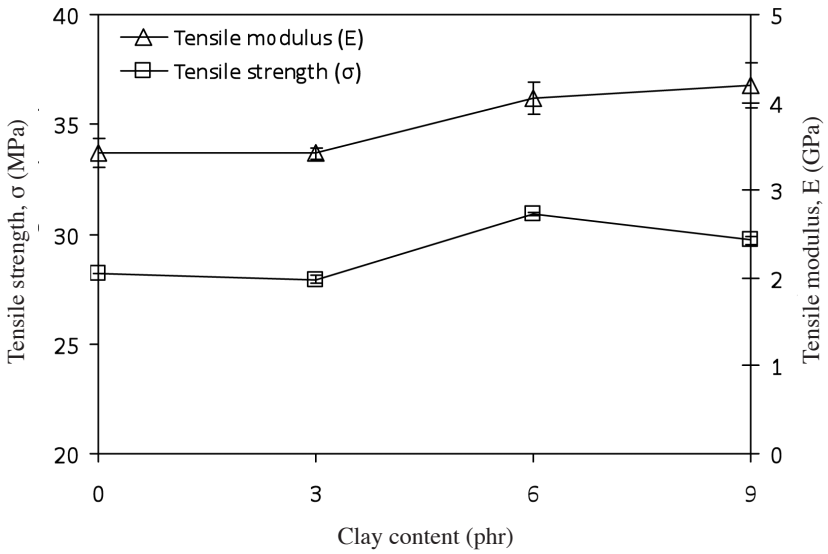

FIGURE 6. Tensile strength and tensile modulus of 15% W_f glass fiber composite with different clay loading

On the other hand, in the tensile results, PP/G15 composite with 6 phr clay gives the highest improvement (Figure 4). As noted earlier, higher filler content (9 phr clay) may result in agglomeration, leading to lower tensile properties.

FLEXURAL PROPERTIES

Flexural strength and modulus of hybrid nanocomposites are shown in Figures 7 and 8. The PP matrix has a flexural strength of 37 MPa and a modulus of 1.0 GPa. In Figure 7, the flexural strengths and moduli of the GF composites containing 0 and 6 phr clay are shown as functions of the fiber weight fraction. From the results, the effectiveness of clay as reinforcing agent is clearly seen. The PP/GF/NC composites, at all fiber weight fractions, have higher flexural properties (strength and modulus), when compared with the flexural properties of the PP/GF composites. Flexural strength increased by 10%, from 37.3 MPa (PP) to 40.8 MPa (PP/NC6) and 23%, from 42.3 MPa (PP/G15) to 52.1 GPa ((PP/G15)/NC6). The flexural strength of (PP/G15)/NC6, is even higher (52.1 MPa) compared with the strength of composite with 30% W_f fiber (PP70/G30) which is 46.9 MPa. In the context of this study, 15% W_f GF composites with 6 phr of NC has higher flexural strength than the composite with higher GF loading, 30% W_f (without NC). As for the composite with higher fiber weight fraction (PP/G30), only a 6% increment in flexural strength value is observed with the incorporation of 6 phr clay into the system ((PP/G30)/NC6). The same behavior has been observed for the flexural modulus (Figure 7). This could be due to the same reason previously discussed for the tensile properties.

Figure 8 shows the effect of clay loading on the flexural properties (strength and modulus) of 15% W_f composites. From this figure, it can be seen that the flexural strength and flexural modulus of PP/GF composites are monotonically increased, with increase in clay loading, being markedly higher than that of the unfilled PP matrix. Although the flexural failure strength of the composite is a fiber dominant property, the matrix has an influence in the overall properties of the composite. Haque et al. (2003) demonstrated that the enhancement in flexural properties of fiber/nanocomposite was achieved due to improved properties of matrix-clay phase composites portion and also unique interfacial fiber-matrix bonding characteristics. Flexural strength of the PP/GF/NC hybrid composites is increased by about 14%, 16% and 40% by the incorporation of 15% W_f GF, 15% W_f GF/3 phr NC, and 15% W_f GF/6 phr NC, relative to the unfilled PP matrix. This is due to the synergistic effects of the GF and NC. Kornmann et al. (2005) reported that the strength of the matrix was improved by the presence of the silicate layer. GF and NC appear to provide a good combination of reinforcements to carry the load during the flexural deformation of composite. However, it is also observed that the optimum flexural strength was achieved at 6 phr clay (52.1 MPa). At 9 phr NC loading, the flexural strength decreases to 51.0 MPa. The distribution of particles in the matrix is an important factor to be considered in this case. The possibility for agglomeration is greater at higher clay content, resulted in the stress concentrating effect. The same behavior has been reported by other researchers (Asi 2009; Chandradass et al. 2008; Kornmann et al. 2005).

The incorporation of clay yields a significant improvement in the flexural modulus of the PP/GF composites, which is attributed to the stiffness and rigidity of the clay nanoparticles (Figure 8). The flexural modulus increased from 1.01 GPa (PP) to 1.91 GPa (87%) and 1.89 GPa (85%) for PP/G15 and (PP/GF15)/NC3 composite, respectively. As for the composite with higher clay loading, 139% increment (to 2.44 GPa) in flexural modulus value is observed with the incorporation of 6 and 9 phr NC into the system ((PP/G15)/NC6 and (PP/G15)/NC9). Clay can adhere on the GF surface as well as on the PP matrix, which affects

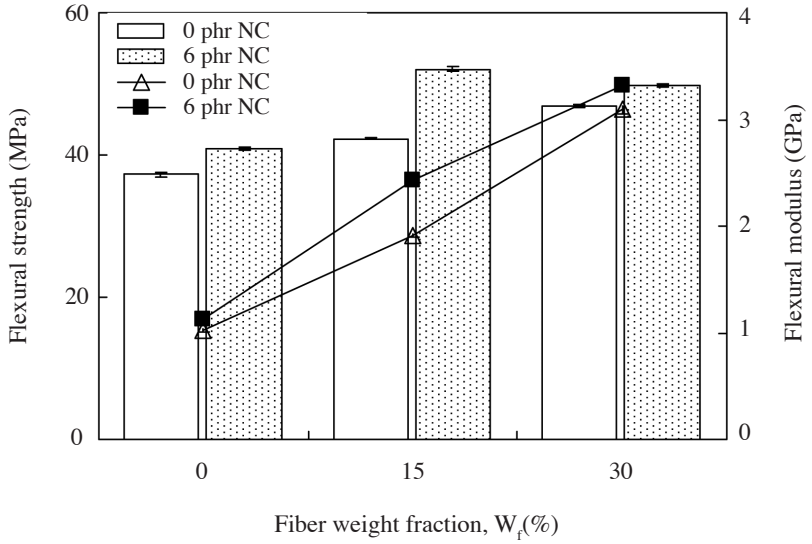

FIGURE 7. Flexural strength and flexural modulus of PP/GF and PP/GF/NC hybrid composites

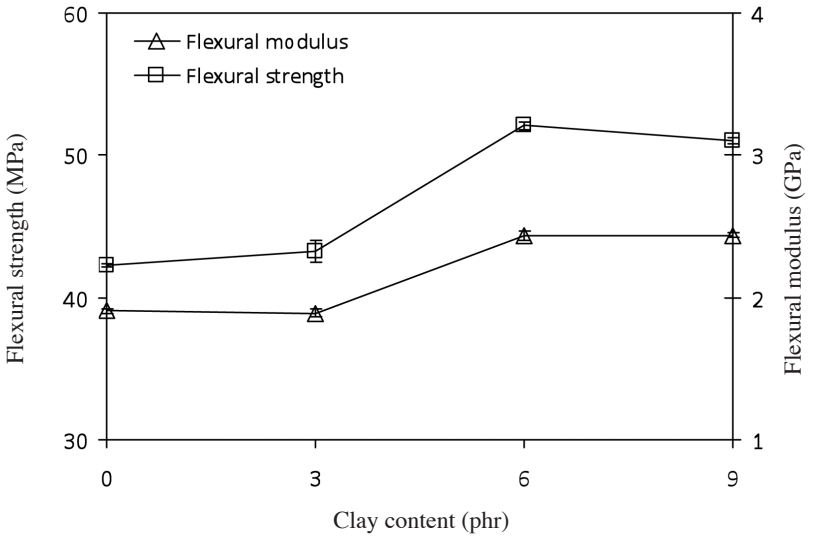

FIGURE 8. Flexural strength and flexural modulus of 15% W_f glass fiber composite with different clay loading

the interfacial properties in the composites such as the adhesive strength and interfacial stiffness of the composite medium (Norkhairunnisa et al. 2007; Wetzel et al. 2002). These two factors play a crucial role in the stress transfer efficiency and the elastic deformation from the matrix to the reinforcement agents. It is probable that this could be due to the surface and contact areas effect. The high surface area of clay increases the contact area with the matrix, thereby increasing the interface. The enhanced interfacial property and effective stress transfer increases the modulus of the fiber composites in nanocomposite.

CONCLUSION

Layered clays were used as nanoparticle fillers in GF reinforced PP composites. The concentration of the particles and their distribution in the matrix were observed to be very important in maximizing the benefits of nanoparticles reinforcement. TEM results revealed NC particle intercalation. The size of the aggregates and the number of platelets in the composites increase with the percentage of clay within the PP matrix. The T_{onset} of PP/GF/NC is seen to have increased by approximately 9°C to 14°C and simultaneously, the initial thermal stabilities at $T_{5\%}$ and $T_{10\%}$ are also observed to be increased up to 56°C and 43°C, respectively, than the conventional PP/GF composite. Tensile strength and tensile modulus of PP/GF composites decreased and increased, respectively, with an increase in fiber loading. The addition of clay nanoparticles improved these properties. Flexural strength and flexural modulus increased with an increase in fiber loading. The addition of clay nanoparticles further improved these properties.

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